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Laser-based instrumentation which employs quasi-optical techniques to measure near millimeter wave dielectric properties has been developed in this laboratory and successfully used to acquire a data base on a wide variety of materials. This report includes the results for various crystalline and non-crystalline solids, semiconductor, and samples of various standard and advanced ceramics. Measurements are at 245 GHz and the results are compared with the data obtained by other researchers on similar samples.

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PRECISION MATERIALS CHARACTERIZATION IN THE NEAR MILLIMETER AND SUBMILLIMETER WAVELENGTH RANGE

FINAL REPORT

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June 1, 1989

U. S. ARMY RESEARCH OFFICE

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ABSTRACT

Laser-based instrumentation which employs quasi-optical techniques to measure near millimeter wave dielectric properties has been developed in this laboratory and successfully used to acquire a database on a wide variety of materials. This report includes the results for various crystalline and non-crystalline solids, semiconductor, and samples of various standard and advanced ceramics. Measurements are at 245 GHz and the results are compared with the data obtained by other researchers on similar samples.

SUMMARY

It has long been known that many important opportunities exist for a mature technology in the near millimeter and submillilmeter spectral region. Among these are such widely varied areas as: short-range secure communications, short-range high-resolution radars, surveillance, diagnostic studies of fusion plasmas, spectroscopy of the gaseous, liquid, and solid states, and studies of combustion, superconductivity, and the Josephson effect. However, many technological challenges must be overcome before systems which operate in this spectral region will become commonplace. Design of passive components such as windows, filters, directional couplers or absorbing coatings is hindered by insufficient information on the dielectric properties of materials. The serious shortage of such data exists particularly in the 100-300 GHz range.

A number of factors may have contributed to the absence of data for this spectral range compared to that which is available for other portions of the electromagnetic spectrum. Certainly, a fundamental problem of measurement technique arises because of the intermediate character of this radiation, which lies between the microwave and infrared spectral region.

Typical microwave measurement techniques which rely on systems using fundamental-mode waveguides, are prohibited by the small dimensions and high losses encountered at these wavelengths. On the other hand, infrared techniques which serve well in the infrared become less suitable because of the relatively long wavelengths which necessitate the use of large apertures and/or corrections for diffraction effects. A simple and versatile instrument[1] designed to

circumvent many of these problems is currently being used in this laboratory to collect data on the optical properties of materials in the near millimeter wave (see Figures 1 and 2). This instrument utilizes the power and spatial coherence of a laser source and combines the use of quasi-optical techniques with the introduction of over-moded dielectric waveguides to limit diffraction spreading of the radiation.

The development of optically-pumped molecular lasers (OPML) has made available both near-millimeter wave (NMMW) and submillimeter wave (SMMW) sources witn ideal properties for material measurements. At the laser operating frequencies, relatively simple apparatus can be used to obtain measurements of high precision. A two-beam instrument assembled in this laboratory has demonstrated the capability for accurate measurement of the refractive index and absorption coefficient for a variety of materials at a frequency of 245 GHz.[1-3]

This report presents the results obtained for the program, "Precision Materials Characterization in the Near Millimeter and Submillimeter Wavelength Range." A substantial database has been collected on a wide range of materials and are tabulated in Tables 1 to 4. This list includes the low-loss, birefringent crystals like, quartz and sapphire. A systematic investigation was carried out on single crystal sapphire of various optical grades. This was aimed at establishing the intrinsic dielectric loss in sapphire and at understanding the mechanisms and processing parameters giving rise to additional extrinsic dielectric loss in ceramic alumina. The best alumina samples have losses approximately 100% larger than sapphire.

Measurements were carried out on sapphire samples of two different optical quality - Hemcore (lower grade) and Hemlite (better grade). Samples were fabricated in sufficiently large sizes to provide the necessary memasurement accuracy. Out of three samples studied, thickness of one was close to 38 mm and the other two were of close to

50 mm each. In addition, one was with the sample face perpendicular to the crystal optical axis (c-cut), and the other two with sample faces parallel to the crystal axis (a-cut). In a-cut sample, a linearly polarized electromagnetic wave normally incident on sample, depending on the rotational orientation of the sample, will have its electric vector aligned parallel or perpendicular to the crystal c-axis. Consequently, use of a-cut samples allows the determinations of the dielectric properties of single crystal sapphire for both orienttations of the electric field. The results obtained at 245 GHz are given in Table 3. So within the quoted experimental uncertainties, the results for the dielectric constant and the loss tangents, and the values for birefringence are virtually identical for the two grades of sapphire.

Quartz is perhaps the premier low-loss material for general purpose usage. The accuracy with which the absorption coefficient can be determined is limited, but the results are consistent with a very low loss on the order 0.01 cm^{-1}. On the other hand, the amorphous form of silicon dioxide, fused silica, has a much higher loss than crystalline quartz. This loss is also sensitive to impurities, especially water content.

Various samples of silicon nitrides, different in terms of their compositions and fabrication procedures, were studied in this program. Silicon nitride has desirable physical and chemical properties for many high temperature, high-strength engineering applications. The values of the dielectric constants at 245 GHz were found to be very close to each other. The measured losses were larger than alumina by an order of magnitude. It shows the need for undertaking an extensive material development program before they can be improved to be comparable to the other candidate window materials.

Other ceramic materials studied in this program include beryllia and boron nitride. The dielectric constants of the beryllia samples

studied were very close to each other. However, their loss-tangent values were very much dependent on their chemical composition and fabrication procedures. Regarding near-millimeter wave dielectric properties, boron nitride appears to be superior to both beryllia and alumina. It is available in a wide range of sizes and shapes, has excellent thermal and mechanical properties, high dielectric strength, and is machinable with conventional tools.

Semiconductors, like silicon, and other samples like, nickel ferrite, also were included in the study. Silicon sample was lightly doped with boron (0.18 ppb) and showed relatively low loss. Whereas, the loss tangent value for the nickel ferrite was relatively large. This may account for much of the insertion loss which is seen in device application of materials of these kinds.

Survey of the data presented below reveals that the dielectric loss is quite variable, even among similar materials. In this spectral range, measuremment uncertainties among nominally identical samples are not uncommon. To circumvent this problem of measurement uncertainties by establishing standards for near millimeter wave dielectric properties, the National Physical Laborotory (UK) and Harry Diamond Laboratories have organized and are conducting an international sample exchange program.[4,5] This laboratory along with several other laboratories from five different countries are participating in this sample exchange program to compare near-millimeter-wave characterization on exactly the same samples. An invited workshop involving the participants to this program was held just prior to the 12th International Conference of Infrared and Millimeter Waves in Orlando, Florida in December 1987.[6] The selected samples and their measurement values obtained by this laboratory are shown in Table 5. Laser-based measurements as such, and the accuracy and consistency of the results were well-received.

CONCLUSION

The database on the NMMW dielectric properties of materials is slowly expanding, primarily by the use of quasi-optical techniques. Results gathered in this program are compared with the data obtained by other researchers on samples similar in nature. Out of all the samples studied, ceramic samples showed considerable variations among samples, prepared by different mamnufacturers or with different methods of preparations, sintering aids used, etc.

Of all the ceramic samples studied, only boron nitride showed low index value, as well as, lowest loss tangent value. The measured loss tangent values for alumina samples was also found to be fairly low. These ceramic materials have potential for successful applications in high-powered gyrotron windows. Any attempt tc improve the performance characteristics of these materials will require to undertake a characterization effort aimed at basic understanding of the parameters and mechanisms controlling the observed dielectric loss. Such a study on various standard and advanced alumina is being undertaken in this laboratory. The various silicon nitride samples studied all showed loss tangents which are significantly higher, almost by an order of magnitude, compared to that of other samples. This suggests that there is a need for an additional development effort before these materials can successfully be used as radomes.

The data reported here were measured at a single frequency; however, they can provide guidance for lower frequency as well. Index values for most materials at NMMW have very weak frequency dependence, while the dielectric loss increases with frequency. Hence, the

absorption coefficients observed at 245 GHz should represent an upper bound to the values at lower frequencies. Extension of this work to other frequencies and temperatures is currently in progress.

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6 ardizing Near-Millimeter Wav^ Dielectric Properties of Some Selected Materials and the Intercomparison of their Measurement Techniques," invited paper presented in a 2-day workshop held in Orlando, FL, 11-12 December, 1987.

FIGURES

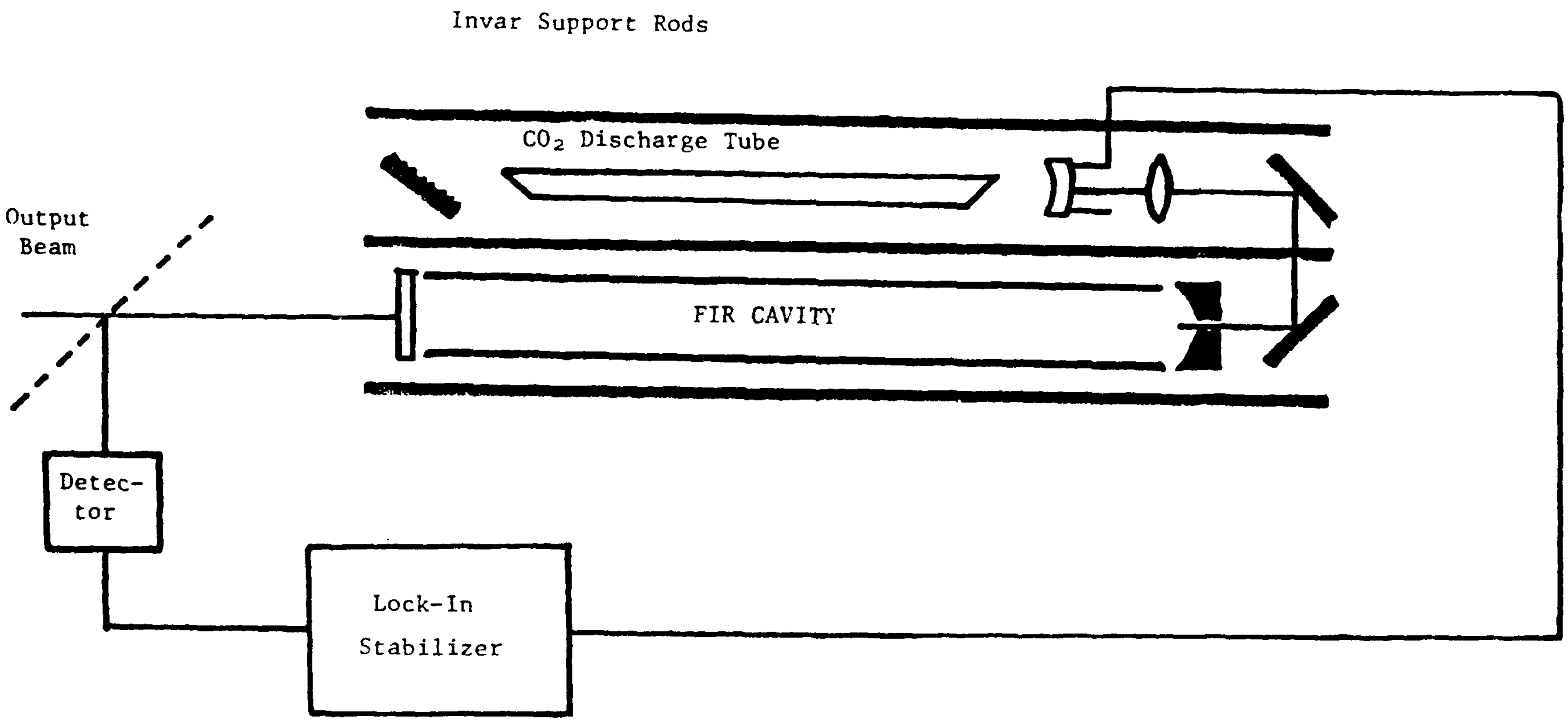

Figure 1. FIR System

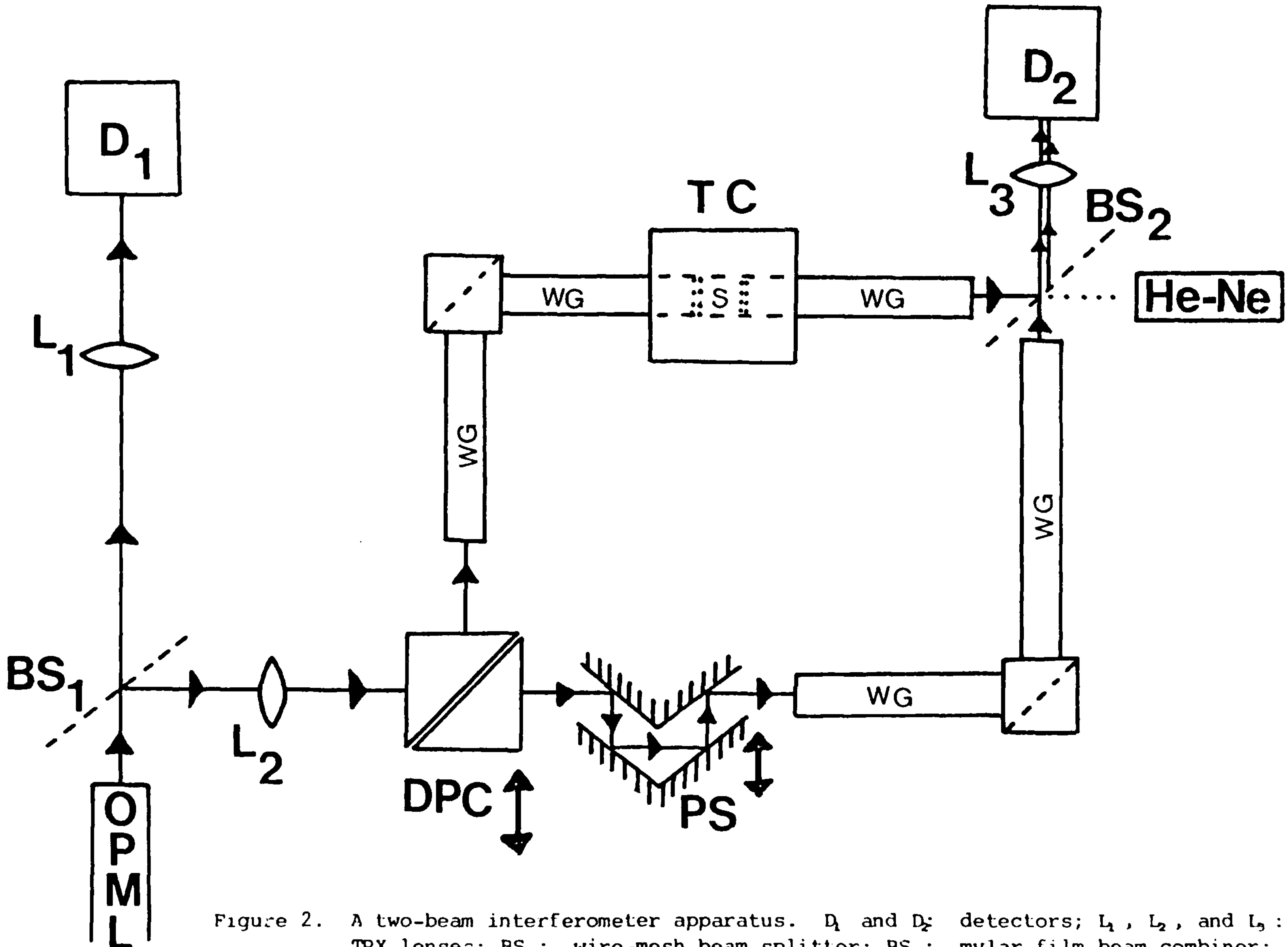

Figure 2. A two-beam interferometer apparatus. D_1 and D_2: detectors; L_1, L_2, and L_3: TPX lenses; BS_1: wire-mesh beam splitter; BS_2: mylar-film beam combiner; DPC: double--prism coupler; PS: phase shifter; S: sample holder; TC: temperature-controlled cell; WG: dielectric waveguides; AL: He-Ne alignment laser; OPML: optically-pumped moledular laser.

TABLES

Table I.

EXPERIMENTAL RESULTS

MATERIAL	This Work at 245 GHz			Literature	
	Thickness	n	$\alpha(cm^{-1})$	n	$\alpha(cm^{-1})$
Rexclite[a] (1422)	56.137 mm	1.5913 ± .0001	0.170 ± .002	1.590 ± .002[31]	0.22[33]
TPX[b]	25.391 mm	1.4576 ± .0001	0.047 ± .003	1.459 ± .001[31] 1.456 ± .002[32]	0.053[33]
Fused[c] Silica	9.990 mm	1.955 ± .001	0.178 ± .009		

a. Polystyrene, cross linked (Obtained from C-LEC Plastics, Inc.).

b. Poly-4-methyl-pentene 1.

c. Dynasil 4000

TABLE 2

EXPERIMENTAL RESULTS

Material	This Work at 245 GHz				Literature	
	Thickness (mm)	n	α (cm^{-1})	Loss Tangent (10^{-4})	n	α (cm^{-1})
Quartz-O	37.145	2.1059 ± .0002	.011 ± .003	1.02 ± .277	2.106[1]	
Quartz-E	37.145	2.1533 ± .0002	.016 ± .006	1.45 ± .542	2.154[1]	
Fused Silica[a]	20.007	1.9516 ± .0001	.81 ± .004	7.97 ± .40	1.95117[2]	0.07[2]
Fused Silica[b]	9.990	1.955 ± .0013	.178 ± .008	17.7 ± .80		
Silicon	10.183	3.4182 ± .0008	.134 ± .016	7.62 ± .91	3.41805[2]	.1303[2]
Beryllia[c]	20.629	2.6126 ± .0003	.100 ± .027	7.44 ± 2.01	2.60846 (44)[3]	.125[3]
Beryllia[d]	25.2623	2.6106 ± 0.003	.182 ± 0.005	13.54 ± 0.37		
Boron Nitride	13.571	2.0727 ± .0004	.069 ± .004	6.38 ± .37	2.05 at 94 GHz[4]	
Nickel Ferrite	12.705	3.7298 ± .0008	.334 ± .016	17.4 ± 1.36	3.73 ± .043[1]	.62[1]

[a] Spectrosil WF [b] Dynasil 4000 [c] National Beryllia Corp. [d] Ceralloy 418S, HP

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[4] W. W. Ho, Report No. AMMRCTR82-28, Army Materials and Mechanics Res. Center, Watertown, MA 02172.

TABLE 3

MATERIAL		Thickness (mm)	This Work at 245 GHz		
			n	$\alpha(cm^{-1})$	Loss Tangent (10^{-4})
Sapphire (Hemlite)[a]	Ordinary	37.89934	3.064195 ± .00017	0.047 ± .009	3.98 ± 0.57
	Extraordinary		3.4030 ± .00016	0.0344 ± .014	1.97 ± 0.80
Sapphire (Hemcore)[b]	Ordinary	50.4980	3.06533 ± .00009	0.0383 ± .02	2.43 ± 1.27
	Extraordinary		3.40406 ± 0.00008	0.0380 ± .02	2.17 ± 1.14
Sapphire (Hemcore)	Ordinary	50.500	3.06555 ± .00012	0.0352 ± 0.02	2.23 ± 1.27
Alumina (Sample #1) Ceralloy 138, HP[c]		25.2349	3.1299 ± 0.0004	0.179 ± 0.023	11.12 ± 1.4
Alumina (Sample #2) Ceralloy 138 HP		12.511024	3.1260 ± 0.001	0.0928 ± 0.01	5.77 ± 0.62
Alumina (Sample #3) Ceralloy 138, HP		12.51610	3.1223 ± .0008	0.0978 ± .016	6.09 ± 1.0

[a]Birefringence is 0.3388.

[b]Birefringence is 0.3387.

[c]Presence of visible secondary phase on surface.

TABLE 4

MATERIAL	Thickness (mm)	This Work at 245 GHz		
		n	$\alpha (cm^{-1})$	$\tan \delta$ (10^{-4})
Silicon Nitride (Sample #1) 1% MgO, HP Ceradyne, Ceralloy 147A	6.1310	2.8924 ± 0.0015	1.003 ± 0.015	67.45 ± 1.01
Silicon Nitride (Sample #2) 1% MgO, HP Ceradyne, Ceralloy 147A	6.1316	2.8984 ± 0.0015	0.950 ± 0.038	63.75 ± 2.55
Silicon Nitride (Sample #5) 1% MgO, HP[a] Ceradyne, Ceralloy 147A	3.1877	2.8457 ± 0.0034	1.59 ± 0.12	108.4 ± 8.2
Silicon Nitride (Sample #4) 8% Y_2O_3, HP Ceradyne, Ceralloy 147Y-1	3.1877	2.8976 ± 0.0027	0.752 ± 0.12	50.50 ± 8.1
Silicon Nitride (Sample #6) 8% Y_2O_3, HP Ceradyne, Ceralloy 147Y-1	5.64007	2.9057 ± 0.0017	0.930 ± 0.07	62.26 ± 4.7
Silicon Nitride Reaction Sintered United Technology	4.47035	2.4260 ± 0.0005	0.33 ± 0.05	26.7 ± 4.0
Silicon Nitride Sintered Y_2O_3, Al_2O_3 United Technology	1.7495	2.9116	0.240	16.05
Silicon Nitride, HP United Technology	6.72645	2.8882 ± 0.0008	1.38 ± 0.043	93.06 ± 2.9

[a]Presence of visible secondary phase on the surface.

Table 5

(NPL Materials)

Material	Thickness (mm)	This Work at 245 GHZ		
		n	$\alpha(cm^{-1})$	Loss Tangent (10^{-4})
Polyethylene	9.946	1.5237 ± .004	0 ± .014	
Rexolite-1	12.42	1.5920 ± .0002	0.145 ± .009	17.7 ± 1.0
Rexolite-2	18.55	1.5930 ± .0004	0.146 ± .008	17.8 ± 1.0
Beryllia	4.95	2.588 ± .001	0.20 ± .04	15 ± 3.0
Macor	1.748	2.391 ± .003	2.89 ± .19	235 ± 15
Quartz-o	7.991	2.1059± .0004	<0.02	
Quartz-e	7.991	2.1551± .0004	<0.02	
Ferroflow	0.437	3.37 ± .15	44 ± 8	2539 ± 461

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